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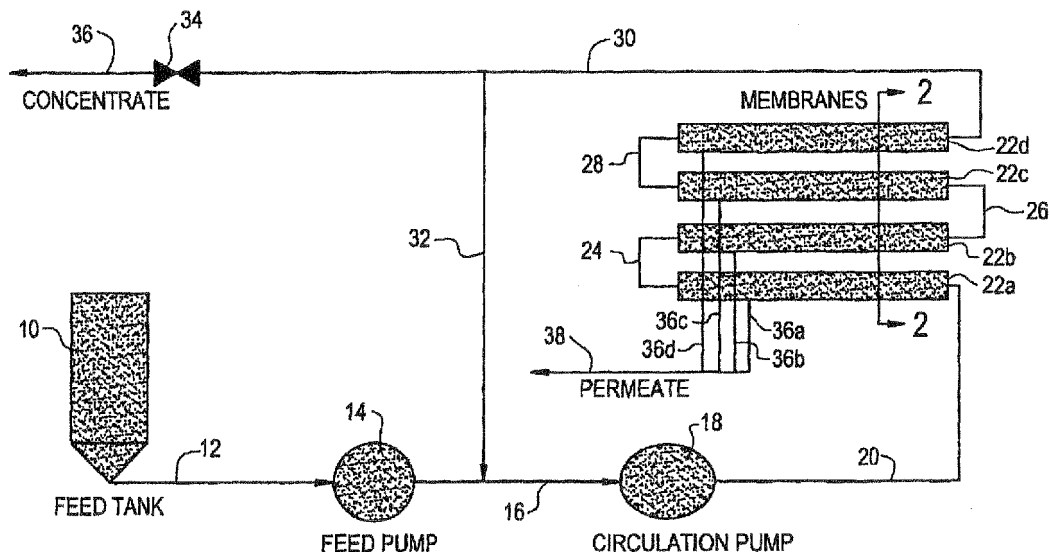
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(54) **Apparatus and process for the separation of liquids and solids**

(57) A liquid solid separation apparatus having a porous metal pipe sealed inside a non-porous metal pipe which allows a portion of the liquids, e.g., hydrocarbons and water to pass through the first pipe into the non porous pipe from which they are removed while solids are retained within the porous pipe. In the process there is

a system pressure which aids in the filtration and a circulating velocity which removes the detained solids. Preferably a portion of these solids with reduced liquid content are recycled back to the system, mixed with fresh feed. By recycling a portion of the recovered solid concentrate the velocity of the flow in the system kept constant and the system itself is stabilized.

FIG. 1



Description

[0001] The present invention relates to an apparatus and process for separating solids, particularly small particulates such as fines from hydrocarbon/water mixtures and in the further separation of hydrocarbon and water.

Related Art

[0002] Some hydrocarbon streams are contaminated with particulate solids. For example, hydrocarbons recovered in soil reclamation under the super fund proceedings. Other examples of hydrocarbon streams which may be contaminated with particulate solids are those recovered in remediation of drilling muds, spent motor oils and the like. Shale oil and oil sand hydrocarbons may also contain small solid particles.

[0003] The solid particles are usually in the range of .1 micron up to 20 and are not easily separated by gravity settling but tend to stay suspended in the hydrocarbons.

[0004] The present invention provides an apparatus and process for separating particulate solids from hydrocarbon stream in a continuous process by pressure filtration of the liquids and cross flow removal of the separated solids from the filter medium.

[0005] Briefly, the present apparatus uses a porous metal pipe sealed inside a non-porous metal pipe which allows the liquids, e.g., hydrocarbons and water to pass from the first pipe in to the second pipe from which they are removed while solids are retained within the first pipe. There is a system pressure which aids in the filtration and a circulating flow which removes the detained solids and preferably recycle a portion of these solids (having greatly reduced liquids content) with fresh feed back to the system.

[0006] The cross flow of the circulating flow prevents buildup of the filtered solids. The hydrocarbons are removed from the chamber formed by the outer pipe. If water is present, the hydrocarbons and water are separated in the outer pipe chamber by decanting, for example, with a bottom drain for the water and an upper drain for the hydrocarbons.

[0007] The term hydrocarbons as used herein includes other organic compounds such as nitrogen, oxygen, sulfur or metal containing organic compounds frequently associated with hydrocarbons.

[0008] FIG. 1 is a schematic representation of one embodiment of the present invention.

[0009] FIG. 2 is a cross section taken along line 2-2 of Fig. 1.

[0010] FIG. 3 is a cross sectional representation of a multiple porous tube configuration.

[0011] Generally, the porous metal component is formed from non-spherical particles which have an irregular shape. These metal particles should have a particle size from 30 to 100 micrometers with from 30 to 40 micrometers being the preferred range. The porous metal component should have pore sizes from 0.5 to 10

micrometers with from 0.5 to 5 micrometers being the preferred range. Generally the porous metal component will have a porosity of 5 to 20% prior to treatment with the metal oxide.

5 [0012] The porous metal component should be formed of a metal which is not corroded by the fluids with which it is intended to be used. Generally austenitic stainless steels are preferred. The particularly preferred stainless steels are the 300 series with 316L being especially preferred.

10 [0013] Preferably the porous metal component in the form of a tube or pipe is coated on the internal surface with a sintered metal oxide.

15 [0014] The metal oxide particles are generally spherical in shape and have a particle size of from 0.2 to 1.0 micrometer. The metal oxide should be sinterable at a temperature below the melting point of the metal used to form the support. Generally the metal oxide should coalesce at below 1200°C. with from 900° to 1200°C. being the preferred range. The preferred metal oxide is titania. The anatase crystalline form of titania which is converted to the rutile crystalline form upon being heated and sintered together has given particularly good results. For titania the sintering temperature should be 20 from 900° to 1200°C. with from 1050° to 1200°C. being preferred.

25 [0015] Preferably the particles removed are larger than about 0.05 micron, preferably about 0.1 micron or larger. Solids larger than about 10 may be more easily removed by other means.

30 [0016] The system is preferably pressured to operate at 10-600 psi. The temperature of the treated materials is adjusted to facilitate the passage of the hydrocarbon through the pores of the substrate and any metal oxide coating by reducing the viscosity. Temperatures in the range of 60-300°F are preferred. The pressure and temperature can be adjusted relative to each other to obtain optimum flow.

35 [0017] An important aspect of the process is the velocity of the material through the inner tube(s) or pipe(s). High velocity removes the solids from the porous metal tube wall and prevents plugging of the pores. The velocity of the feed and recirculation material is that sufficient to remove the solids from the inner tubes(s) or pipe(s), preferably about 10 to 20 feet per second. The preferred velocity is 15 feet per second of the material in the system, i.e., fresh feed and recirculated material passing through the porous inner tube(s) or pipe(s). Feed rate of material to the circulating system may 40 range from 1 to 2100 gallons per minute.

45 [0018] The apparatus is comprised of one or more, preferably multiple, porous metal tubes and more preferably a plurality of such tubes running through and enclosed by a non-porous container, e.g., a larger pipe which forms a chamber to receive the filtered liquid.

50 [0019] A preferred system is comprised of one or more stainless steel tubes 6-75 mm in diameter with a TiO₂ coating sintered to the inside diameter. Typically

the tubes are welded together into an all stainless steel membrane module. One or more modules may be connected in series or parallel.

[0020] The hydrocarbon feed material may contain water in addition to the solid particles. Up to 25 vol.% water may be present in the fresh feed.

[0021] Referring now to Fig. 1 a particular embodiment is illustrated as a schematic flow diagram. A hydrocarbon material containing particulate solids preferably in the range of .1 to 20 microns is accumulated in a feed tank 10 from which it passes via line 12 to feed pump 14 which serves to pressurize and supply feed to the system. From the feed pump the pressurized feed passes via line 16 to circulation pump 18 which maintains the velocity of the material flowing through the separating zone. The feed enters the separating zone through line 20. In this embodiment the separating zone is comprised of multiple sections, 22a, 22b, 22c and 22d, arranged in series. In section 22a, the hydrocarbon feed flows through porous metal tube 40a, where a portion of the liquids in the feed (hydrocarbons and optionally up to about 0-25 vol percent water) pass through porous tube 40a into the chamber 21a formed by tube 22a surrounding porous tube 40a. The porous tube must be enclosed by an outer non-porous tube or some equivalent structure to create a chamber to receive the permeated liquid. The permeated liquid is collected in the chamber 21a and exits via line 36a.

[0022] In a similar manner the feed stream flows out of section 22a through regular non-porous conduit 24 into section 22b where a further portion of the liquid permeates porous metal tube 40b into chamber 21b from which it passes via line 36b. From section 22b via line 26 the feed stream passes to section 22c where a further portion of the liquid permeates porous metal tube 40c, is collected in chamber 21c and removed via line 36c. The material in section 22c passes via non-porous tube 28 to section 22d where a final portion of the feed liquid passes into chamber 21d and is removed via line 36d. Each permeate removal line feeds to a single line 38 for recovery.

[0023] The solid concentrate exits the treatment separating zone via line 30 from which it may be removed from the system through valve 34 and line 36 or all or a portion may be returned to the hydrocarbon feed to the separation zone. Alternatively the recycled concentrate may be returned to the feed tank. The recycle concentrate is considered as feed component for determining the necessary feed velocity through the system and is used to keep the feed at a constant solids density above the source hydrocarbon, thereby optimizing the system and allowing constant operating conditions for the most economic and effective operation. Any number of porous metal tubes may be positioned within a chamber and any number of sections may be arranged in series in the apparatus to achieve the degree of separation desired.

[0024] The concentrate may be used as bunker fuel

or in the case of a hydrocarbon source derived from a remediation system, the concentrate may be returned to the remediation system. If the permeate recovered via line 38 contains water, the stream may be sent to a distillation unit (not shown) or decanter (not shown) to separate water and hydrocarbons.

[0025] Fig. 3 illustrates an embodiment which is particularly useful for hydrocarbon/water/solid streams. There are four porous metal tubes, 60a - 60d, positioned within non-porous tube 60 either in a system as shown in Fig. 1 or other configuration. The hydrocarbon and water permeate the porous tubes and water is removed via line 66 while hydrocarbons are removed from chamber 63 via line 64.

EXAMPLE

[0026] The source feed was derived from a hydrocarbon vapor recovery system used to purify vapor from a drilling mud remediation in which diesel boiling range material used as a component of the drilling mud. The source hydrocarbon feed had the characteristics shown in the TABLE.

TABLE

Hydrocarbons	85 Vol. %
Water	5 Vol. %
Solids	10 Vol. %
Particle size range	.1 to 20 microns

[0027] In an apparatus arranged similarly to that illustrated in FIG. 1, the source feed was pumped from the feed tank into the separation zone at a pressure of 25-300 psig, temperature of 150°F and at a rate of 5-10 gpm. Each section of the separation zone comprises four 5 ft. long, 1/2 inch ID stainless having titanium dioxide internal coating and positioned centrally as shown in FIG. 3 and sealed inside a 5 ft. long, 2 1/2 ID stainless steel pipe. A portion of the solid concentrate was recycled to the feed line, as required, to maintain the system velocity.

Claims

1. An apparatus comprising:

a non-porous tube, at least one porous metal tube positioned in said non-porous tubular member, and sealed within said non-porous tube, said non-porous tube having an upper portion and a lower portion;
at least one drain line in said non-porous tube;
a feed line connected to said tubular porous metal tube,
a solids removal line connected to porous metal tube distal to feed line; and

a circulating cross flow pump operably positioned on the feed line.

2. The apparatus according to claim 1, wherein said porous metal tube is coated with a sintered metal oxide. 5
3. The apparatus according to claim 2, wherein said porous metal tube is coated internally with said sintered metal oxide. 10
4. The apparatus according to claim 3, wherein said metal oxide comprises titanium oxide.
5. The apparatus according to claim 3, wherein said porous metal comprises stainless steel. 15
6. A process for the separation of solids from hydrocarbons comprising: 20
feeding a stream containing liquid hydrocarbons and particulate solids in a first ratio of solids to liquids to a porous tubular metal zone, under pressure conditions and at a velocity to prevent solids from adhering to the porous metal structure, 25
separating a first liquid portion from the solid particles by passing said first liquid portion through said porous metal zone into a nonporous tubular zone surrounding said porous tubular structure, 30
recovering said first liquid portion from said nonporous tubular zone and
passing a second liquid portion containing said particulate solids in a second ratio of solids to liquids from said porous tubular zone. 35
7. The process according to claim 6, wherein said pressure is in the range of 10 to 600 psi. 40
8. The process according to claim 6, wherein said solid particles are larger than about 0.05 microns.
9. The process according to claim 6, wherein a portion of said second liquid portion containing said particulate solids is recirculated to said porous tubular zone. 45
10. The process according to claim 6, wherein fresh feed is added to said process at a rate of from 1 to 2100 gallons per minute. 50

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FIG. 1

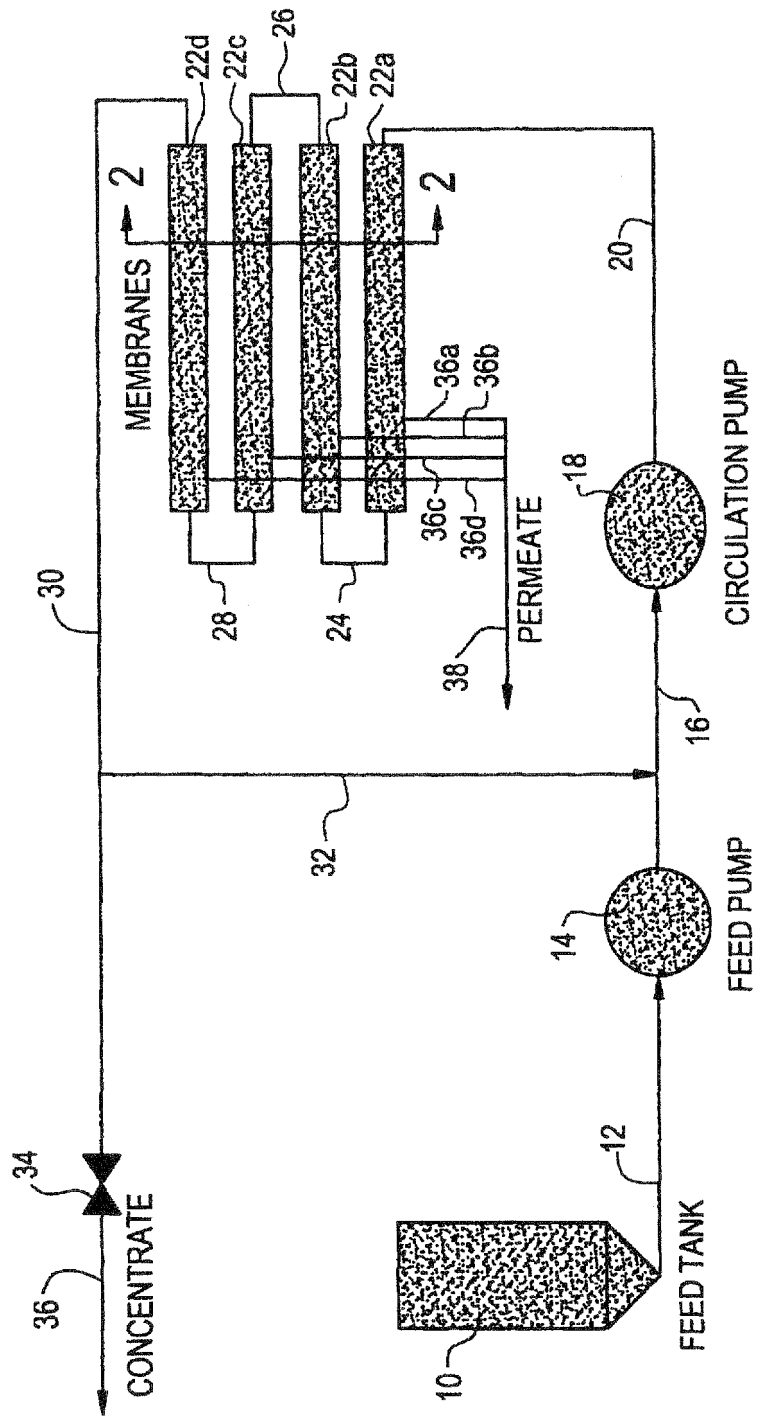


FIG. 2

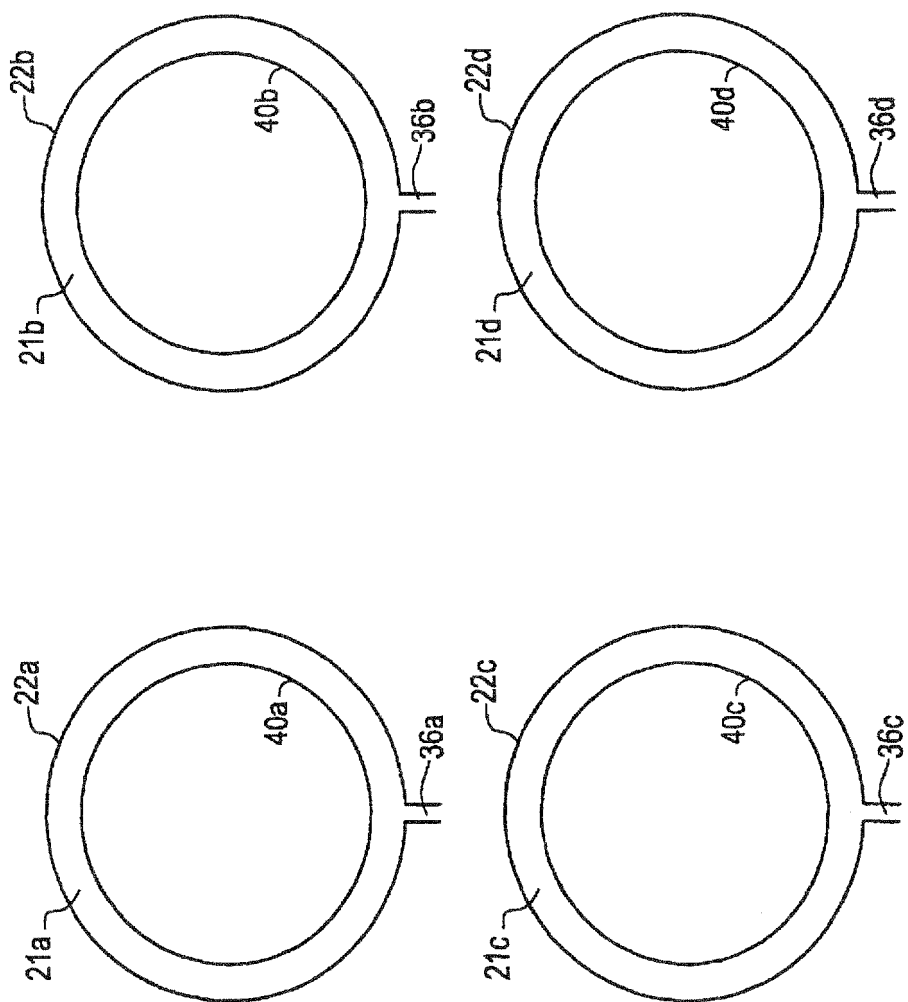
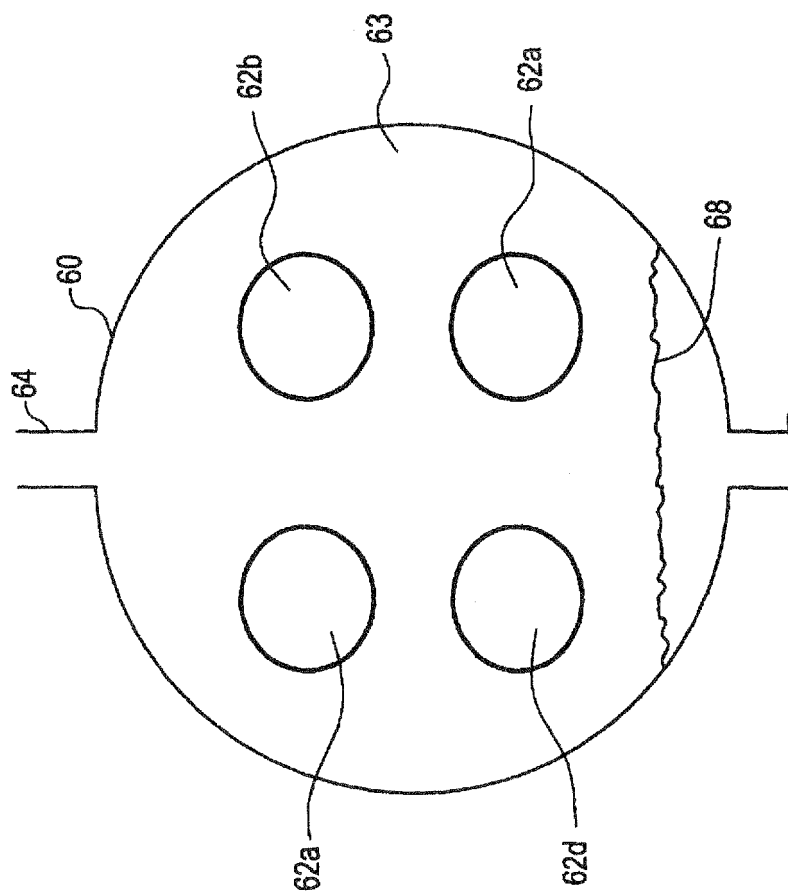


FIG. 3





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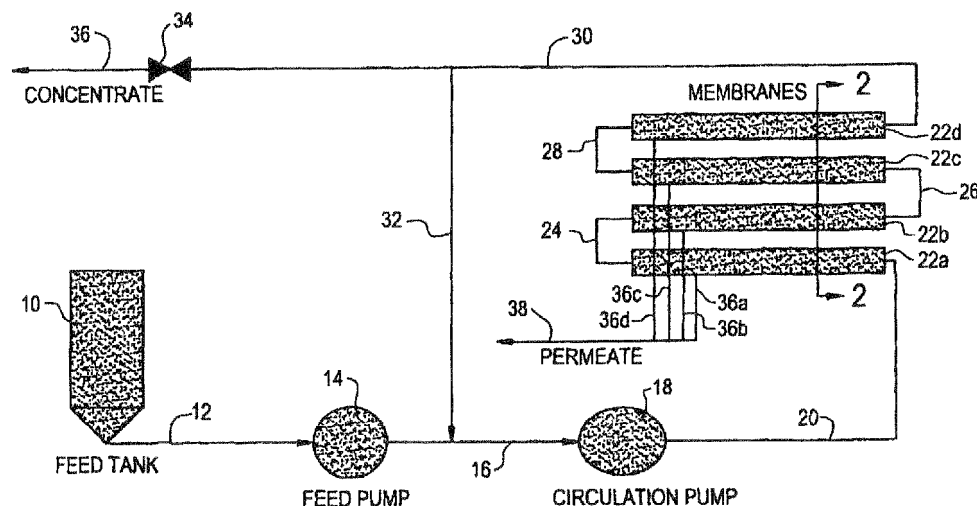
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FIG. 1





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Office

EUROPEAN SEARCH REPORT

Application Number
EP 99 30 0656

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US 5 149 447 A (MASHAYEKHI MANSOUR ET AL) 22 September 1992 (1992-09-22) * abstract; figures 1-3 * * column 2, line 11 - line 21 * * column 2, line 55 - line 62 * * column 3, line 9 - line 18 * * column 4, line 37 - column 5, line 48 * * column 6, line 61 - column 7, line 4 * * column 7, line 42 - line 50 * * column 8, line 29 - line 60 * * example 1 *	1,5-10	B01D63/06 C10G31/09 C10G31/11 B01D61/14
X	US 4 411 790 A (AROD JEAN ET AL) 25 October 1983 (1983-10-25) * abstract; figures 1,2 * * column 1, line 9 - line 15 * * column 1, line 64 - column 2, line 50 * * column 3, line 13 - line 68 * * column 4, line 12 - line 26 * * example 2 *	1-4,6-10	
X	US 5 074 989 A (SIGAUD JEAN B ET AL) 24 December 1991 (1991-12-24) * abstract; claims 1-12; figures * * column 1, line 13 - line 27 * * column 1, line 40 - line 48 * * column 2, line 4 - line 46 * * column 2, line 61 - column 3, line 44 * * column 3, line 58 - column 4, line 19 * * column 4, line 38 - line 58 * * column 5, line 2 - line 9 *	1-4,6-10	TECHNICAL FIELDS SEARCHED (Int.Cl.6) C10G B01D
X	US 4 865 742 A (FALLETTI FLORENCE) 12 September 1989 (1989-09-12) * the whole document *	1-4,6-10	
-/--			
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 8 November 1999	Examiner Hoornaert, P
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

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European Patent
Office

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Application Number
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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	US 4 186 100 A (MOTT LAMBERT H) 29 January 1980 (1980-01-29) * the whole document *	1,6	
A	---	6-10	
X	US 4 200 533 A (BRANDON CRAIG A ET AL) 29 April 1980 (1980-04-29) * the whole document *	1,6	
A	---	6-10	
X	US 4 372 859 A (SUGIMOTO YOSHIKAZU ET AL) 8 February 1983 (1983-02-08) * abstract; figures 1-3,6 * * column 3, line 32 - column 7, line 17 *	1,6	
A	---	6-10	
A	EP 0 381 812 A (DU PONT) 16 August 1990 (1990-08-16) * the whole document *	2-5	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 8 November 1999	Examiner Hoornaert, P
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	
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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 99 30 0656

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08-11-1999

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 5149447	A	22-09-1992	CA 2075531 A,C	08-02-1994
US 4411790	A	25-10-1983	FR 2482975 A	27-11-1981
			AT 15067 T	15-09-1985
			CA 1168590 A	05-06-1984
			EP 0041013 A	02-12-1981
			ES 502439 A	01-11-1982
US 5074989	A	24-12-1991	FR 2587629 A	27-03-1987
			AT 47329 T	15-11-1989
			EP 0216711 A	01-04-1987
			JP 2107132 C	06-11-1996
			JP 8016227 B	21-02-1996
			JP 63122788 A	26-05-1988
US 4865742	A	12-09-1989	CA 1334938 A	28-03-1995
			EP 0332788 A	20-09-1989
			AU 604303 B	13-12-1990
			AU 1386688 A	23-11-1995
US 4186100	A	29-01-1980	US 4088576 A	09-05-1978
			DE 2820057 A	22-11-1979
			FR 2424756 A	30-11-1979
			GB 1593127 A	15-07-1981
			JP 1359106 C	13-01-1987
			JP 54146076 A	14-11-1979
			JP 61020327 B	21-05-1986
US 4200533	A	29-04-1980	NONE	
US 4372859	A	08-02-1983	JP 1344590 C	29-10-1986
			JP 56026503 A	14-03-1981
			JP 61008729 B	17-03-1986
EP 0381812	A	16-08-1990	US 4888114 A	19-12-1989
			AT 156382 T	15-08-1997
			AU 613789 B	08-08-1991
			AU 4128689 A	16-08-1990
			CA 1325389 A	21-12-1993
			CN 1044768 A,B	22-08-1990
			DE 68928237 D	11-09-1997
			DE 68928237 T	12-03-1998
			IL 91578 A	31-07-1994
			IN 172057 A	27-03-1993
			JP 2233117 A	14-09-1990
			JP 2903128 B	07-06-1999

EPO FORM P459

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